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Crystal and Vibrational Structure of Energetic 3,5-dinitro-1,3,5-oxadiazinane (DOD) by Single Crystal X-ray Diffractometry and Raman Spectroscopy

by Joseph E Banning, Kristopher D Behler, and Rosario C Sausa

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14. ABSTRACT <p>A prerequisite for establishing structure-property-performance relationships of materials is the understanding of their structures and crystal packing. Here, we report an isolation procedure for producing high-quality crystals of energetic 3,5-dinitro-1,3,5-oxadiazinane (C₃H₆N₄O₅) and our studies on its room temperature crystal structure and packing by single crystal X-ray diffraction and its vibrational modes by Raman spectroscopy. The title compound consists of 2 nitrate groups attached to a chair-like hexagonal ring containing 3 carbon atoms, 2 nitrogen atoms, and an oxygen atom. Both nitrate groups adopt axial positions with respect to the ring and the dihedral angles between the nitrate groups, and the plane containing the 3-ring carbon atoms are 64.93 (9)° and 60.55 (9)°, similar to those of α-RDX involving its axial nitrate groups. Contacts between the oxygen and hydrogen atoms of adjacent molecules dominate the intermolecular interactions. Based on the title compound's unit cell constants, we determine a density of 1.732 g/cm³ at 298 K, which agrees well with the value of 1.699 g/cm³ obtained by quantum mechanical calculations using a temperature of 298 K.</p>					
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1. Introduction

The physical and chemical properties of 3,5-dinitro-1,3,5-oxadiazinane (DOD) make it a promising energetic material with potential use as a TNT replacement in melt-castable explosives. DOD exhibits a calculated density and heat of formation (ΔH_f) of 1.699 g/cm³ and -22.213 kJ/mol, respectively, at 298 K and a measured melting point of approximately 376 K.¹⁻⁴ It possesses good thermal stability, as it decomposes above 473 K, with 98% conversion to products at 483 K,²⁻⁴ and good sensitivity to impact, as indicated by electrostatic potential map calculations.¹ Chute et al. and Tabouis, Ortigues, and Auberstein discovered DOD as a byproduct during the synthesis of RDX circa 1950, and Ishchenko et al. reported its synthesis from methylene dinitramide in 1996.⁵⁻⁹ More recently, Li et al. isolated DOD during the synthesis of RDX, less than 1% by mass, and reported its crystal structure at 103 K.^{4,10}

Figure 1 presents the molecular structure of the DOD and RDX compounds. A DOD molecule consists of a heterocyclic, 6-membered ring with 5 alternating carbon and nitrogen atoms and 1 oxygen atom between 2 of the carbon atoms. The molecule contains 2 nitro groups (-NO₂), each bonded to 1 of the 2 nitrogen ring atoms, and 3 pairs of hydrogen atoms, each bonded to 1 of the carbon ring atoms. The structures of DOD and RDX are nearly identical, except that an oxygen atom in the DOD ring replaces one of the N-NO₂ groups in the RDX ring. In DOD, the 2 nitro groups stand on the same side of the ring (axial position), whereas in the most stable RDX polymorph (α -RDX), 1 nitro group adopts an equatorial (E) position while the other 2 groups adopt axial positions (A) (equatorial, axial, axial (EAA) conformation).^{4,10-12}

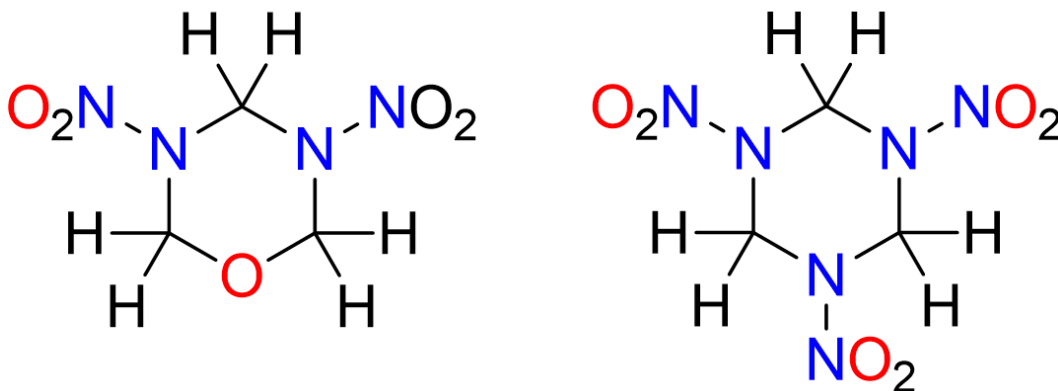


Fig. 1 Structural formulas of DOD (left) and RDX (right)

In this report, we describe an isolation procedure to produce hundreds of milligram-quantities of DOD and confirm its identity by single crystal X-ray diffractometry and Raman spectroscopy. We determine and report its key intermolecular interactions, crystal packing, and vibrational features and compare and contrast them to those of α -RDX. Further, we determine its crystal density at room temperature and compare the result to the theoretical value determined at 298 K.

2. Experimental Procedures

2.1 Synthesis

Crude DOD was obtained using the method of Ishchenkoiv et al.^{7–9} Paraformaldehyde (0.9 g, 0.03 mol) and N,N'-dinitromethylenediamine (1.36 g, 0.01 mol) were added with vigorous stirring to a mixture of 2 mL 90% sulfuric acid and 20 mL of methylene chloride at 283 K. The mixture was stirred for 5 h at 293 K. The organic layer, separated from the mixture by decanting, was washed with water (5×5 mL) and then dried over MgSO_4 at room temperature for several hours. Crystalline DOD was obtained by removing the volatiles by rotary evaporation at room temperature and dissolving the remaining residue in 3:1 isopropanol:water by heating. The hot solution was placed in the freezer and allowed to solidify. Then, the frozen mixture was warmed to room temperature, at which point 65 mg of DOD was collected by suction filtration. DOD was recrystallized twice, first by dissolving it in hot chloroform and then by slowly evaporating the solvent over the course of several days. This procedure yielded clear, high-quality crystals for X-ray analysis.

2.2 Characterization

X-ray Crystallography. DOD crystals were characterized with a SuperNova, Dualflex, EosS2 diffractometer using a $\text{Mo K}\alpha$ ($\lambda = 0.71073 \text{ \AA}$) radiation source and an EosS2 charged coupled device (CCD) detector at 297.93(10) K (Rigaku–Oxford, Japan). CrysAlis PRO and mercury were used in data collection, cell refinement, and data reduction.^{13–15} The molecular structure was determined with SHELXT using intrinsic phasing and refined with SHELXL using least squares minimization.^{16–18} The hydrogen atoms were refined using a riding model with $\text{C–H} = 0.97 \text{ \AA}$ and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. Table 1 lists the crystal data, data collection, and structure refinement details.

Table 1 Experimental crystallographic details for DOD

Molecular and unit cell information	
Crystal data	Value
Empirical formula	C ₃ H ₆ N ₄ O ₅
Formula weight	178.12
Crystal system	monoclinic
Space group	P2 ₁ /n
a/Å	5.8390(4)
b/Å	17.6086(10)
c/Å	7.1309(5)
α /°	90
β /°	111.267(8)
γ /°	90
Volume/Å ³	683.25(8)
Z	4
ρ_{calc} /g/cm ³	1.732
μ /mm ⁻¹	0.163
F(000)	368.0
Crystal size/mm ³	0.5 × 0.6 × 0.5
Data acquisition details	
Collection data	Value
Temperature/K	297.93(10)
Radiation	MoK α (λ = 0.71073)
2 Θ range for data collection/°	4.626 to 52.722
Index ranges	-7 ≤ h ≤ 7, -22 ≤ k ≤ 21, -8 ≤ l ≤ 8
Reflections collected	5711
Structure refinement	
Refinement statistics	Value
Independent reflections	1400 [R _{int} = 0.0307, R _{sigma} = 0.0261]
Data/restraints/parameters	1400/0/110
Goodness-of-fit on F ²	1.033
Final R indexes [I ≥ 2 σ (I)]	R ₁ = 0.0374, wR ₂ = 0.0889
Final R indexes [all data]	R ₁ = 0.0463, wR ₂ = 0.0946
Largest diff. peak/hole / eÅ ⁻³	0.15/-0.16

Raman Spectroscopy. DOD spectra were acquired using a Horiba LabRAM HR Evolution (Horiba Jobin Yvon, Kyoto, Japan) spectrometer, configured with a 600 l/mm grating, 150 μ m hole, and 5% neutral density filter. The data were collected in a backscattering geometry using a Lexel SHG (Cambridge Lasers

Laboratories, Inc, Fremont, California) ion laser tuned to 514 nm. We attenuated the 1-W laser beam to less than 5 mW with a 5% neutral density filter to prevent possible sample decomposition. The Horiba's LabSpec 6 (version 6.4.2.5) software was used to collect the data over the range of 50–4000 cm^{-1} . Scans were performed using 10 accumulations of 5 s while utilizing the built-in spike filter to remove cosmic rays. A silicon wafer served to hold the sample and to check the calibration of the instrument by using the Si peak at 520.7 cm^{-1} as a reference.

3. Results and Discussion

3.1 Molecular Structure

Single crystal X-ray diffraction and structure analysis of DOD revealed its molecular conformation at room temperature. The conformation is presented in Fig. 2. There are no unusual bond lengths and angles, and our values agree overall with those measured previously for DOD at 103 K.^{4,10} The N1–N2 and N3–N4 bond distances measure 1.396 (2) and 1.393 (2) Å, nearly identical with the two axial N–N bonds of α -RDX, whose average value equals 1.395 (3) Å at room temperature.⁹ The hexagonal ring adopts a chair-like conformation, where the N1, C2, C3, and N3 atoms lie in a plane [root-mean-square deviation (RMSD) = 0.0082 (2) Å] and the NO₂ groups adopt axial positions. In this conformation, the shortest distance from the C1 atom to the plane N1–C2–C3–N3 measures 0.548 (2) Å, whereas the shortest distance from the O1 atom to the plane measures –0.650 (2) Å. The atoms of the N–NO₂ groups are nearly planar [RMSD = 0.013 (5) and 0.011(1) Å] for the respective N1–N–O–O and N3–N–O–O planes, and their centroid-to-centroid distance measures 1.937 (1) Å. The dihedral angles between the C1–C2–C3 plane and the N1–NO₂ plane or N3–NO₂ plane are 64.93 (9)° and 60.55 (9)°, respectively, similar to those involving the axial N–NO₂ planes of α -RDX (~62°).¹¹

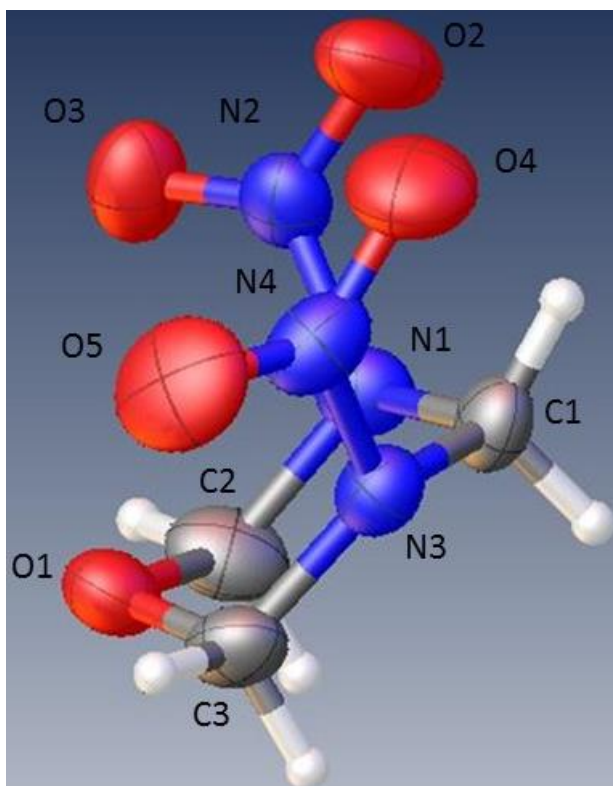


Fig. 2 Molecular conformation of DOD. Nonhydrogen atoms are depicted as 50% probability displacement ellipsoids.

Raman spectroscopy provides DOD vibrational structural information. Figure 3 reveals the Stokes features resulting from the inelastic scattering of a DOD crystal with 514 cm^{-1} radiation. The features reflect the molecule's modes of vibration. We tentatively assign the prominent features based in part on those reported previously for RDX.^{19–22} The peaks near 100 and 125 cm^{-1} result from lattice motions, whereas the peaks near 230 and 260 cm^{-1} result from ring out-of-plane bending vibrations. The peaks near 430 cm^{-1} and the one near 550 cm^{-1} result from ring in-plane bending. The strong peak near 880 cm^{-1} is a key signature for many energetic NO_2 -containing compounds and results from C–N–C ring stretching. In contrast, the peaks near 886 and 920 cm^{-1} result from N– NO_2 stretching and in-plane CH_2 bending. The peak near 1300 cm^{-1} results from N– NO_2 stretching, whereas the peak near 1600 cm^{-1} is attributed to asymmetric NO_2 stretching. The strong features in the region between 2900 and 3100 cm^{-1} result from C–H asymmetric stretching vibrations. We searched for reported features associated with C–O–C vibrations in the regions between 800 and 860 cm^{-1} and 1000 and 1200 cm^{-1} , but were unable to identify them unequivocally.^{23–26} These features are Raman-insensitive and/or overshadowed by other peaks in our spectrum, unlike the infrared-sensitive DOD C–O–C stretching vibration at 1183.2 cm^{-1} reported by Li et al.¹⁰

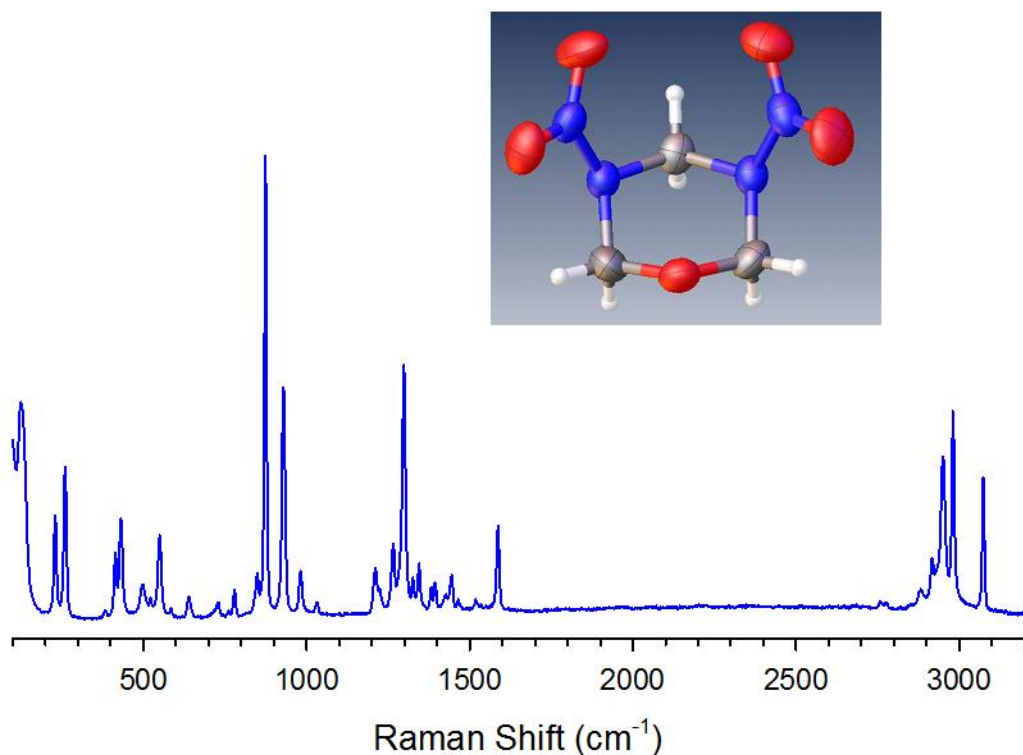


Fig. 3 DOD Raman spectrum in the regions between 100 and 3200 cm⁻¹. The inset shows the structure of DOD; the C, H, N, and O atoms are depicted in grey, white, blue, and red, respectively.

3.2 Intermolecular Interactions and Density

Figure 4 shows the intermolecular interaction of DOD. Strong bifurcated contacts between the O2 and H atoms of adjacent molecules [$\text{O2}\cdots\text{H2A}^i = 2.520(2) \text{ \AA}$ and $\text{O2}\cdots\text{H3B}^{ii} = 2.596(2) \text{ \AA}$]; symmetry codes (i) $-\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}+z$; (ii) $1-x, 1-y, 1-z$], as well as contacts between the O4 and H atoms of adjacent molecules [$\text{O4}\cdots\text{H3A}^i = 2.602(2) \text{ \AA}$; symmetry code (i) $-1+x, y, z$] dominate the intermolecular interactions and contribute to its stability. The molecules stack themselves in intertwining rows nearly along the *a* axis with an interatomic separation of $5.846(3) \text{ \AA}$. The O1 and C1 atoms of each molecule in a particular row position themselves in an opposite direction from the corresponding atoms of molecules in the adjacent row, resulting in intermolecular O1–O1 atom separations of 3.193 and $3.287(2) \text{ \AA}$ (Fig. 5).

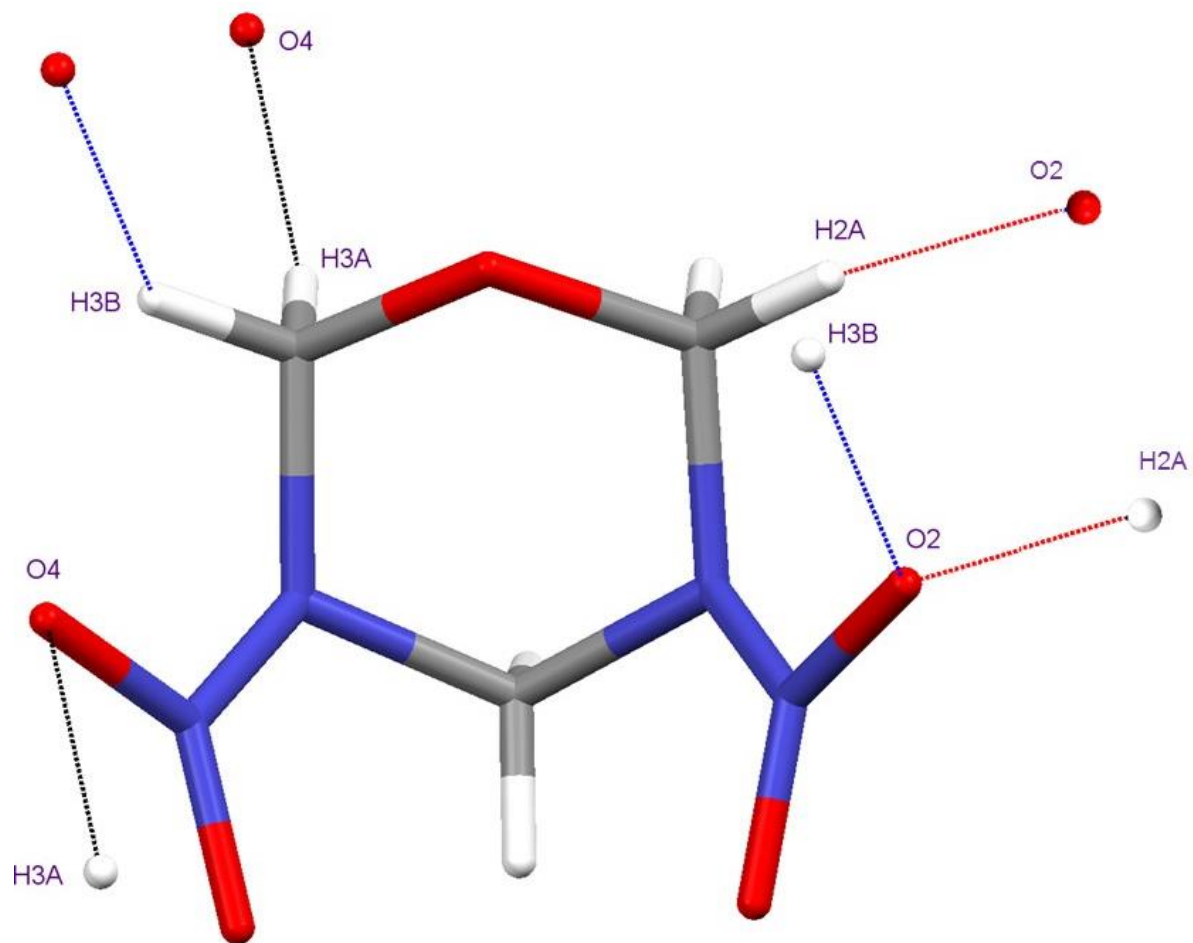


Fig. 4 DOD molecule revealing its intermolecular interactions, represented as dashed lines. The C, H, O, and N atoms are depicted in grey, white, red, and blue, respectively, whereas the bond colors are based on the color of the atoms.

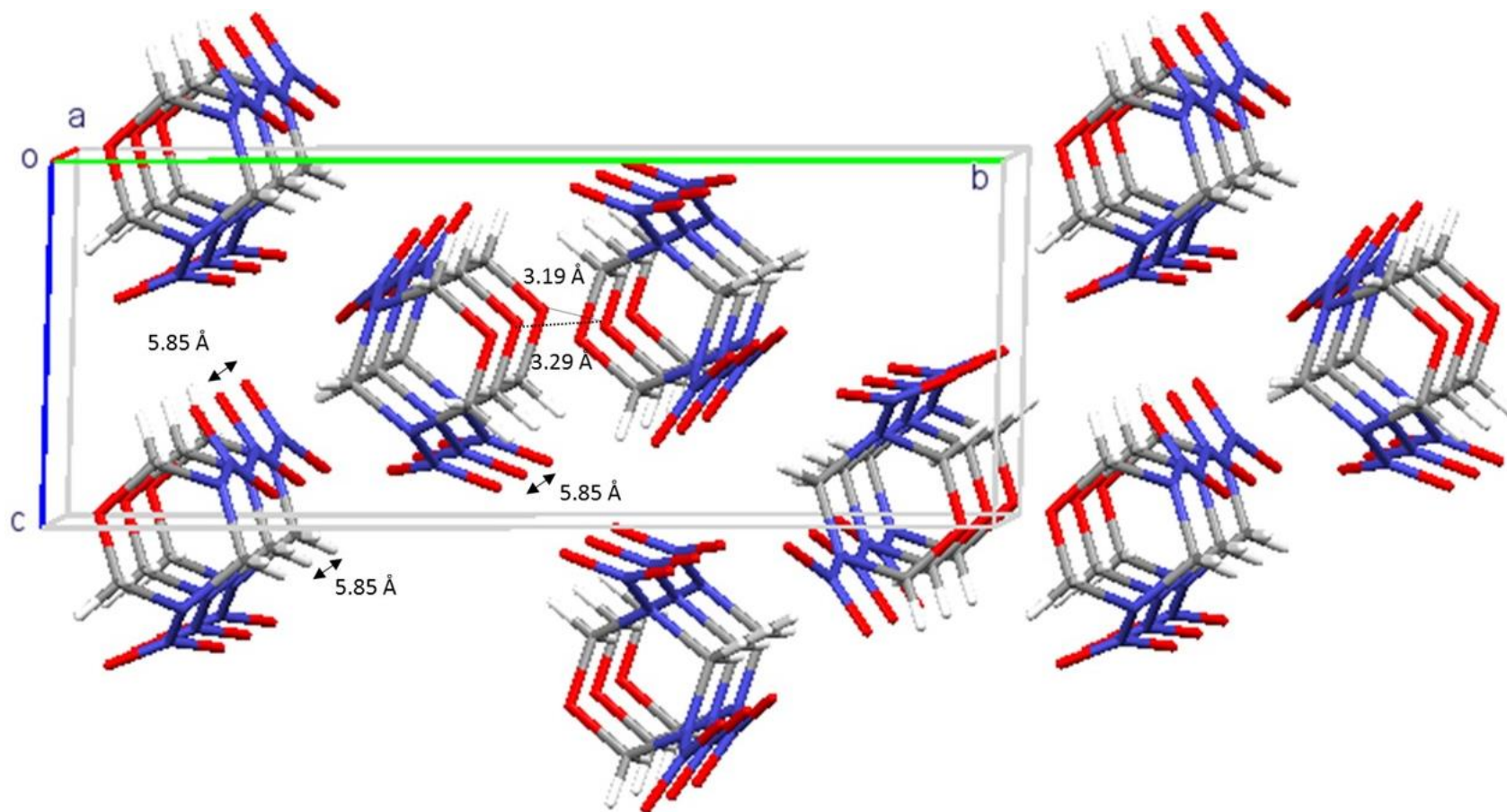


Fig. 5 Crystal packing of DOD. The N, O, and H atoms are depicted in blue, red, and white, respectively. Strong O···H interactions are not shown for simplicity.

DOD belongs to the monoclinic crystal system, space group $P2_1/n$, as revealed by its crystal structure. Its unit cell constants measure a (Å) = 5.8390(4), b (Å) = 17.6086(10), c (Å) = 7.1309(5), $\alpha/^\circ = 90$, $\beta/^\circ = 111.267$ (8), and $\gamma/^\circ = 90$ at 297.9 (1) K, which are similar to those determined previously for DOD at 103 K.^{4,10} Based on the cell dimensions and the molecular weight of DOD, we determine a crystal density of 1.732 g/cm³ at 298 K, which is in accordance with the value of 1.792 g/cm³ reported for 103 K and agrees well with the theoretical value of 1.699 g/cm³ obtained by quantum mechanical calculations using a temperature of 298 K.

4. Conclusions

In this work we produced high quality X-ray diffracting DOD crystals and determined the molecular structure of DOD by both single crystal X-ray diffraction and Raman spectroscopy at room temperature. Overall, the values of the bond angles, lengths, and unit cell constants are in accordance with those measured previously at 103 K. Short contacts between the oxygen atoms of the nitrate groups and the hydrogen atoms of adjacent molecules (~2.52 to 2.60 Å) result in strong intermolecular interactions, which govern the crystal packing, stability, and density of DOD. Prominent Raman features near 880 cm⁻¹ (C–N–C ring stretching), 920 cm⁻¹ (N–NO₂ stretching and in-plane CH₂ bending), 1300 cm⁻¹ (N–NO₂ stretching), and in the region between 2900 and 3100 cm⁻¹ (C–H asymmetric stretching) dominate its Raman spectrum. Our experimentally determined density agrees well with the calculated density (1.732 vs. 1.699 g/cm³ at 298 K). DOD's stability and density make it an attractive energetic material for future applications.

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List of Symbols, Abbreviations, and Acronyms

ARL	US Army Research Laboratory
CCD	charged coupled device
DOD	3,5-dinitro-1,3,5-oxadiazinane
EAA	equatorial, axial, axial
RDX	1,3,5-trinitro-1,3,5-triazine
RMSD	root mean square deviation
TNT	2,4,6-trinitrotoluene

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